

TEST EQUIPMENT AND TECHNIQUES FOR EVALUATING
COMPOSITE PROPELLANT PROCESSING AND HANDLING HAZARDS

by

V. T. Dinsdale¹

THIOKOL CHEMICAL CORPORATION
Wasatch Division
Brigham City, Utah

ABSTRACT

An essential aspect of rocket propellant manufacturing is the accurate evaluation of processing and handling hazards. To determine the hazards of propellant constituents, intermediate compositions, and final compositions present in composite solid propellant, adequate test equipment and techniques were required. The program initiated by Thiokol Chemical Corporation's Wasatch Division to develop the specialized test equipment and techniques necessary will be the subject of this paper.

Hazards were evaluated for normal process conditions and for hazardous conditions which could result from equipment failure or operator error. The primary objective of the program was to develop equipment and techniques with procedures requiring minimum manpower and equipment expenditures.

Equipment was developed to obtain hazard data for friction, impact, thermal, and electrostatic sensitivity. Preliminary work was also conducted to determine the detonation properties of propellants using small laboratory samples.

INTRODUCTION

With rapid advances being made in the development of higher energy propellants, the demand for hazard test data for new propellant constituents, intermediate, and final compositions has also increased. These data are often required prior to making laboratory samples larger than 10 grams because of personnel exposure during some handling and manufacturing operations. Lack of a means to rapidly determine this information with small laboratory samples reduces operating efficiency due to delays incurred while waiting for the test data and the excessive time required to make the number of test samples.

¹Supervisor, Explosive Development Laboratory

Most of the effort expended during recent years on hazard test equipment improvement programs was on the development of precise hazard measuring equipment. The major portion of this equipment was unsatisfactory for production-type testing, because the equipment is not capable of testing minimum size test samples in a short period of time.

The purpose of the program, as summarized in this paper, was to improve existing hazard test equipment designs and test techniques to permit more efficient hazard determination using minimum sample sizes.

INVESTIGATION

A survey was made of Thiokol Chemical Corporation Wasatch Division propellant processing facilities to determine the type and extent of hazardous environments that exist during normal operations or that could exist as a result of minor equipment failure or operator error. This information was classified into types of hazardous environments and the physical state of materials that would be present in these processing environments, i.e., powders, liquids, or solids. Hazardous environments given major consideration were friction, impact, thermal, electrostatic, and detonation. Materials currently being processed were thoroughly evaluated; and with existing facility design and safety practices, these materials do not pose unnecessary hazards to processing equipment or to personnel. However, during normal operations a certain amount of hazardous environments exist. New compositions which may be more sensitive and create a hazardous condition during processing must be evaluated. All combustible materials, including fuel and oxidizer powders, pre-mixes, intermediate propellant compositions, and uncured and cured propellants, are evaluated with this equipment to simulate the various process conditions.

Estimated energy levels for friction, impact, and heat were obtained primarily by duplicating operating and minor incident conditions. A study of types of equipment necessary to duplicate these environments was then made to ascertain the most severe operating hazards present during normal operations and minor failures and also to establish a hazard limit.¹

A survey was then made of several propellant and explosive manufacturing facilities in the United States to determine availability of equipment that could be used to obtain the required hazard data. This equipment also was to comply with the program objectives of being able to obtain this information with procedures requiring small test samples and minimum manpower and equipment expenditures. A summary of the information obtained from this survey and the resultant equipment designed during this program is as follows.

¹Hazard limit is the environment that will barely cause the test material to react.

Friction Testing

A review of the friction equipment used throughout the propellant and explosive industry showed that there was only one satisfactory friction tester design available. This piece of equipment was a strip friction tester designed by Allegany Ballistics Laboratory. However, this tester would meet only part of the test requirements. The design was more than adequate, but Thiokol ordnance designers felt that the same data could be obtained with a tester less expensive to fabricate and operate. Available friction testers of designs that were used for a number of years were not considered satisfactory because of unrealistic friction values and difficulty of operation.

Three basic friction testers were subsequently designed. The first was a slurry friction tester, which with minor changes, was converted into three separate testers to duplicate all friction environments and permit testing of the various types of materials. One of the testers uses a one inch diameter piston and cup arrangement similar to that shown in Figure 1. The applied forces are shown in Figure 2-C. The piston is lowered on top of the sample and into the cup prior to starting the motor. The force is varied by adding weights to the piston carriage, and the rotating speed is adjusted by changing pulley ratios. This type of tester was not considered an accurate friction measuring device; however, the tester does provide an evaluation of the friction sensitivity of materials that might be subjected to confined friction environments, such as combustible materials in a mixer packing gland.

The second slurry friction tester utilized a twelve inch bowl with a spring-loaded friction head that impinges against the side of the bowl. The tester and the forces involved are shown in Figures 2-A and 3. The applied force and rotation speed are set by changing the spring tension on the friction-head assembly and changing the pulley ratios. The test material is distributed in front of the friction head by two Teflon directional scoops. Medium-viscosity materials such as intermediate and uncured propellant compositions are the only materials tested with this device because the tester scrapes on a vertical plane. This tester simulates friction environments experienced in mixing and cleanup operations. Various types of friction head and bowl materials can be used to achieve the desired friction environment. The vertical tester was considered in addition to the horizontal scraper because of the constant test radius eliminating the changing force variable.

The third type of slurry friction tester also utilized a twelve inch bowl, but the friction head applies force against a bottom portion of the bowl that is grooved to contain low viscosity liquids. The friction tester and the application of forces are shown in Figures 2-B and 4. The force on the friction head is controlled by adding or removing weights from the top of the assembly. Friction conditions are simulated for mixing and cleanup processes involving low-viscosity premixes and intermediate and uncured propellant compositions. Friction heads and bowl materials are changed to achieve the desired friction environment.

A strip friction tester was designed for testing fine powders and propellant samples. Two thin strips of metal similar to those shown in Figure 5 are used for each test. The strip surfaces are machine cut to eliminate surface variables. The tester and the application of forces are shown in Figures 6 and 7. Obtaining the desired force to the roller compressing the strips together is accomplished by removal or addition of weights on a lever. A falling weight provides the energy to pull the strips apart. The falling weight impinges upon a lever attached to a wheel, which in turn pulls one of the strips. The other strip is secured to the stand. The applied force and the mass of the falling weight are varied to obtain the desired friction environment. The direct impact tester was modified to incorporate this device as an attachment. The strip tester also used the same framework and drop weight as the direct impact tester.

This particular test not only provides a means of determining friction sensitivity of powder materials and propellant samples that is difficult to obtain on other types of friction testers, but also duplicates friction environments occurring in processes such as scraping of a drum on a floor contaminated with oxidizer and propellant scraps.

A third type of friction tester called, a rotary friction tester, was designed (Figure 8). A rotating wheel impinges upon a shoe of known surface area to produce the friction environment. This tester is primarily used to determine friction sensitivity of viscous materials (premix and uncured propellants). Tests are conducted on cured propellants, but the setup time is excessive. The applied force is varied by adding or removing weights on the end of a lever attached to the friction shoe (Figure 9). The wheel speed is varied by a variable-speed gear box and pulley system. Shoe and wheel materials are changed to obtain any desired combination.

Impact Testing

Prior to initiation of this program, Thiokol's Wasatch Division ordnance engineers designed an impact tester based upon test and design data obtained from the Naval Ordnance Laboratories at Silver Spring, Maryland. Throughout the missile industry there are numerous impact-tester designs, very few of which are directly comparable. Even when designs are the same, sample preparation and test techniques are different. The Thiokol tester was designed to reduce as many of the test variables as possible and improve on test efficiency. The design consisted of a piston and cup arrangement, with the piston guided into the cup as shown in Figures 10 and 11. Propellant samples are prepared by cutting thin slices using a microtome cutter (uncured samples are frozen with CO_2 prior to slicing). Powdered samples are weighed out or measured volumetrically. A disc of sandpaper is placed face down into the sample to increase sensitivity. To avoid excessive galling of the striker surface, a thin shim of steel, 0.005 inch thick, is used between the striker surface and the sandpaper disc.

The second impact tester is called a direct impact tester (Figure 12) because the striker impacts directly on the sample as shown in Figure 13. A small sample of controlled size is placed on the anvil (Figure 14), and the anvil is inserted into the anvil holder and impacted by the falling weight. The drop height and weights are changed to obtain the desired impact environments.

Autoignition Testing

Autoignition tests are conducted in a convective-type oven because most auto-ignition data are required on samples too large for a Woods' metal bath arrangement. Depending upon whether the samples are liquid or solid the samples are suspended in the oven as shown in Figures 15 and 16. A thermocouple is suspended adjacent to the sample to record the sample temperature during test and to indicate when the sample fires. The oven is designed to withstand the firing of up to ten gram propellant samples and contains a blowout plug and exhaust ducting to prevent contamination of the surrounding areas.

Electrostatic Testing

Most of the electrostatic test devices used by the various facilities were not permanent and lacked unity of design. Several of the designs used common devices for energy application, such as the use of a phonograph needle for one electrode, but the devices still varied in sample size, and sample preparation. It was decided to design an electrostatic tester that would incorporate many of the better features of existing designs to meet program objectives. The electrostatic tester and the major subassemblies are shown in Figures 17, 18, and 19. A sketch of the sample test fixture is shown in Figure 20. Samples are inserted into a plastic holder of a given hole size and then placed on a steel blank attached to one electrode. The other electrode is attached to a steel ball placed on top of the hole in the plastic holder. The ball is four times the diameter of the sample hole.

The capacitors are charged with a NJE high voltage power supply capable of generating 60,000 volts. Switching is accomplished with a solenoid operated knife switch to eliminate switch bounce experienced with vacuum switches. Arrangement of the capacitors in a circle with the contacts in the center permits rapid selection of the desired capacitance value.

Detonation Testing

The most important detonation characteristic in propellant processing facilities is the critical diameter.¹ If a critical diameter exists within the maximum diameter of material being processed, then other detonation characteristics such as minimum

¹Critical diameter is the minimum charge diameter that will sustain a detonation when initiated by a booster charge greater than the minimum booster or equal in diameter to the test charge.

booster, deflagration-to-detonation, and projectile impact sensitivity will be determined.

After reviewing other facilities, it was found that very few improvements were made in the method of determining critical diameters, and that all attempts to determine critical diameters of compositions with subcritical diameter samples were unsuccessful.

Two approaches to determine critical diameters with subcritical diameter charges are being investigated. The first is the development of a relationship between the critical diameter of a material and grain reaction determinations extrapolated to a theoretical detonation temperature and other chemical and physical parameters such as density, temperature, and confinement. The second method is a study of the detonation reactivity of subcritical diameter propellant samples when subjected to large booster charges.

INTERPRETATION

The following is a summary of the test techniques and data interpretation for each of the testing devices described.

Friction Analysis

One Inch Diameter Piston-to-Cup Slurry Tester--The most uniform results were obtained using a sample size of approximately 0.4 gram so the material would not be extruded out of the cup. A vertical force and rotational speed were obtained for which a reference uncured propellant sample would fire in approximately one minute. A fire is evidenced by an audible or visual reaction. If the cup or piston temperature rises above 10 degrees Fahrenheit during a test, cooling is provided to maintain a temperature within 10 degrees of room temperature prior to the next test. Ten tests were run on each sample to obtain the minimum, maximum, and average fire times.

A sensitivity index is established by dividing the average fire time of the test material by the average fire time of the reference material and multiplying by ten. Hazard limits are established based on the index values.

Twelve Inch Diameter Vertical Slurry Friction Tester--A thin film of propellant is spread around the periphery of the bowl the same width as the friction head. The sample sizes vary between 0.5 and 2 grams. On some tests it is desirable to put the test sample in front of the friction head instead of spreading it around the side of the bowl. The speed and force are varied to obtain a fire point for a reference material of approximately 15 seconds. A fire is evidenced by an audible or visual reaction. The temperature of the bowl and friction head is maintained to within 10 degrees of ambient temperature prior to each test. One test replaceable metal strips are used on the friction head when conducting standard tests to determine the friction index.

The number of tests conducted and the evaluation of the test data for establishment of a friction index are the same as on the piston-and-cup tests.

Twelve Inch Diameter Horizontal Slurry Friction Tester--The horizontal friction tester uses a sample size of approximately 5 grams. The rotational speed and force settings, number of tests, and data evaluation are similar to the vertical tester.

Strip Friction Tester--The most uniform test results were achieved by placing the sample between the strips in front of the roller pressure point. The preparation of the sample and uniformity of the strips must be carefully monitored to produce accurate test results.

A standard is tested with either beta-HMX (Cyclotetramethylenetetranitramine) explosives or a propellant composition, depending upon the type of test material. The most useful information is obtained by determining a drop height and weight to provide satisfactory results with the standard. Then all similar materials are tested using the drop weight and height, with the applied force being varied.

A Bruceton-type analysis is followed to obtain a 50 percent fire point. The 50 percent fire point is used to establish a friction index, as compared to a reference material. A fire is evidenced by an audible or visual reaction.

Rotary Friction Tester--The rotary friction tester is used for two types of tests. One test is to establish a rotary friction sensitivity index using a stainless steel shoe and wheel. The other is a comparative test using varying shoe and wheel combinations similar to that shown in Figure 21.

The average time-to-fire is used on the standard test to compute a rotary friction index, which is the average time-to-fire of the test material divided by the average time-to-fire of the standard material multiplied by ten.

On the tests using varying wheel and shoe combinations, the data are evaluated by plotting the average time-to-fire in seconds versus the radial wheel velocity in feet per second times the applied force in pounds per square inch (Figure 22). By this method, varying wheel and shoe sizes are used and still compared with other tests using different size wheels and shoes.

Impact Analysis

Indirect Impact Tester--Preparation of propellant samples for the indirect impact tester is accomplished by slicing the propellant into 0.021 inch thick strips using a microtome cutter. Dried materials are sampled volumetrically. The Bruceton test technique is used to obtain the 0, 50, and 100 percent fire points, with the 50 percent fire point being used for determining the impact index. Beta-HMX is

used as a standard; and the reference material, an uncured propellant composition, is used to compute the impact index. The impact index is computed similarly to the rotary friction index, or the 50 percent fire point of the unknown material is divided by the 50 percent fire point of the reference uncured propellant multiplied by ten. A typical test data sheet is shown in Figure 23.

Direct Impact Tester--Because of the difficulty or time consuming efforts in raising or lowering the weights on the direct impact tester, a Probst analysis is used rather than the Bruceton analysis. The Probst analysis involves conducting 20 tests at each height within the range of the 0 and 100 percent fire points. Small (0.009 gram) samples are used. Propellants are sliced with a microtome cutter and punched out with a circular punch. An audible sound is used to determine whether or not the material fired.

Autoignition Analysis

Autoignition tests are conducted with the oven set at a constant temperature. A thermocouple placed adjacent to the sample records the temperature and the time-to-fire. The temperature is increased or decreased until the autoignition characteristics for approximately one hour are obtained, similar to the data shown in Figure 24. If a material to be tested has an unknown autoignition temperature range, a dynamic temperature environment test is performed on the material with the oven temperature increased over a given period of time until the fire point is reached. These data are then evaluated to determine the temperature range to be used.

Electrostatic Analysis

Electrostatic tests are conducted between the ranges of 100 pf to 0.1 μ f capacitance and 100 to 60,000 volts. A 50 percent fire point is estimated using a Bruceton-type analysis by increasing or decreasing the voltage. A chart for which voltages, capacitance, and energy levels are displayed is shown in Figure 25. The sensitivity of materials is compared by taking a constant energy line in joules which is tangent to the test data curve. This energy value is then used to compute an electrostatic sensitivity index, the same as for the friction and impact test data.

Use of the one test replaceable plastic holders enables rapid testing and uniformity of sample preparation with a minimum material required.

Detonation Analysis

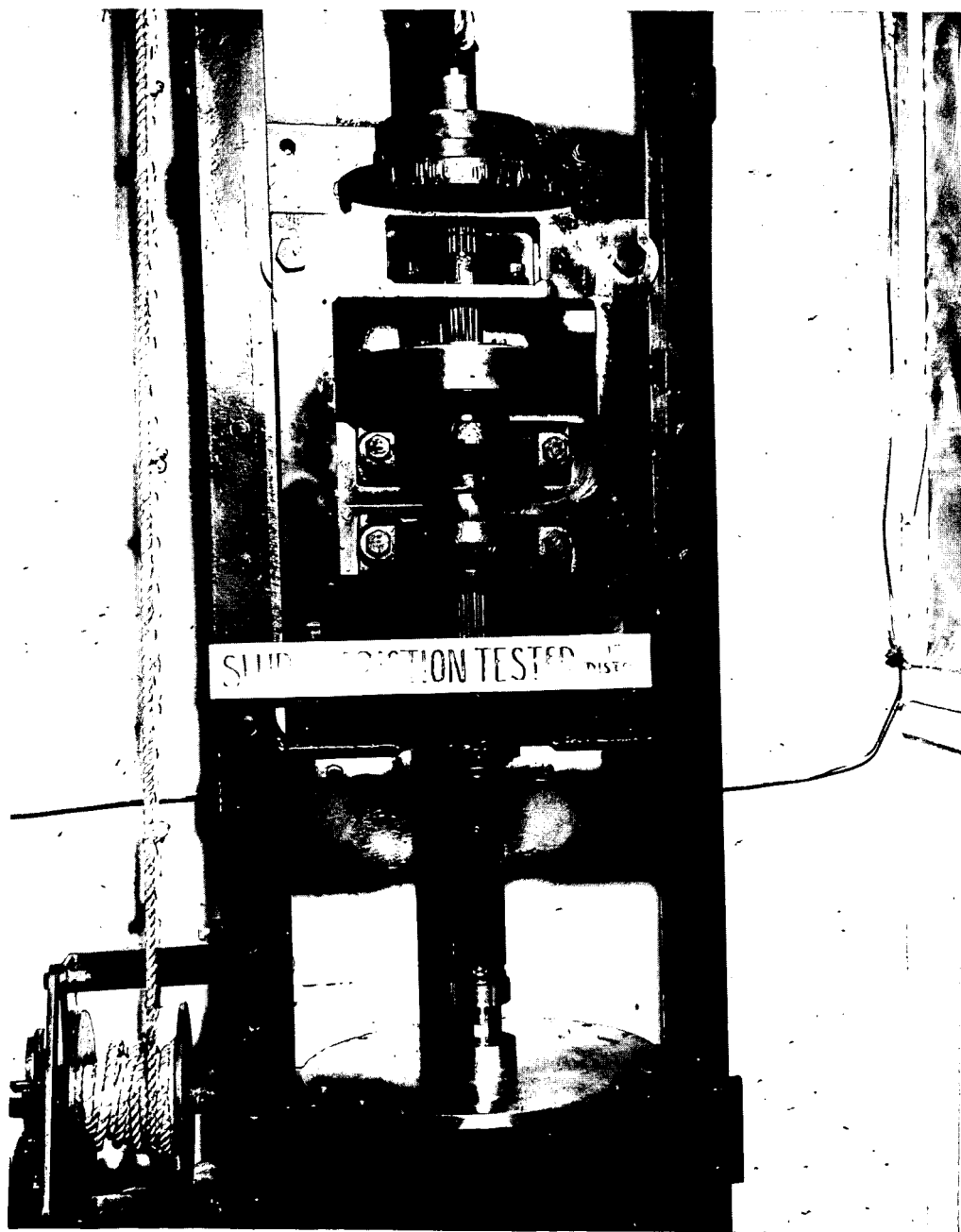
At the present time, sufficient tests had not been conducted to prove the validity of determining critical diameters either by reaction rates or reactivity measurements with laboratory samples.

Tests are still being conducted using a standard pipe detonation test setup similar to that shown in Figure 26. A booster having the same diameter as the test charge is used to ensure adequate boosting, and a charge of sufficient length is used to evidence the reaction. Pin gages are used to record the shock velocities, and the signals from the gages are recorded with a coded pin mixer system to ensure more reliable data pickup.

CONCLUSION

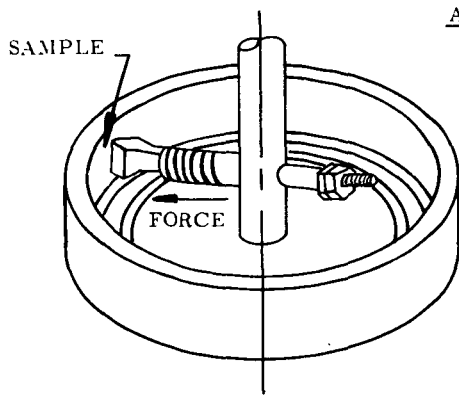
The friction, impact, autoignition, and electrostatic test equipment and techniques developed during this program enable rapid and inexpensive determination of the hazardous characteristics for propellant constituents and compositions present in propellant manufacturing and storage facilities.

This equipment and these techniques are providing better utilization of propellant development facilities and manpower because hazard test data are being obtained with less test delays than was possible with previous designs and techniques.

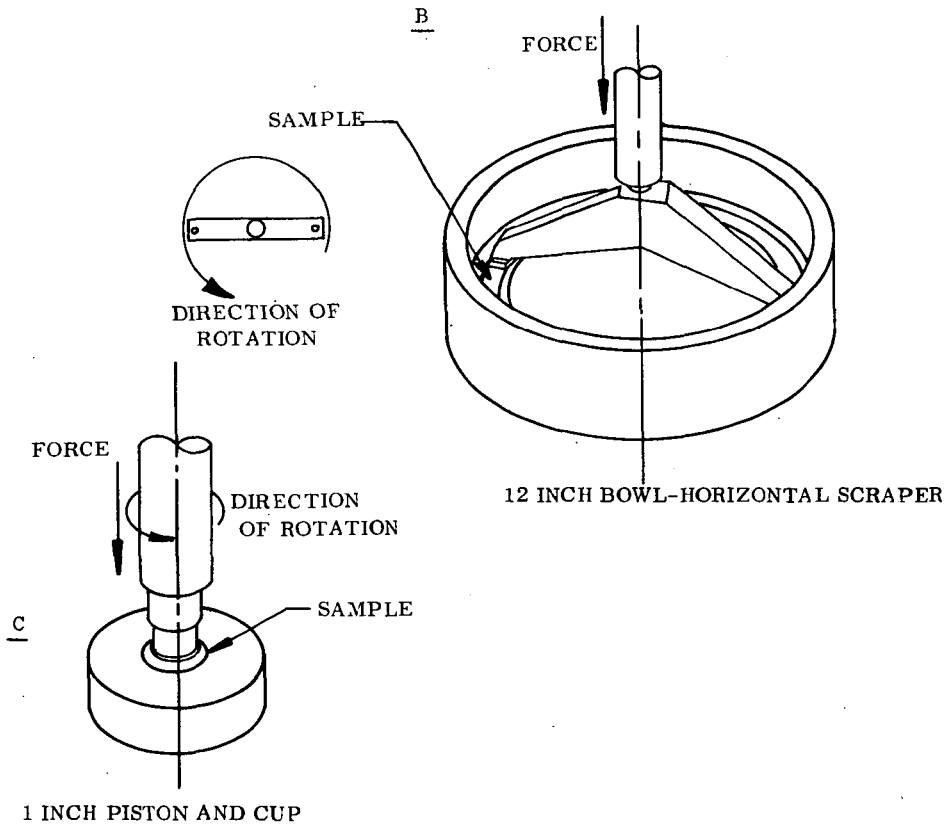


Thiokol CHEMICAL CORPORATION
WASATCH DIVISION • BRIGHAM CITY, UTAH

Figure 1. One Inch Piston and Cup Slurry Friction Tester



12 INCH BOWL-VERTICAL SCRAPER



1 INCH PISTON AND CUP

Figure 2

Application of Forces in Slurry Friction Testers



Thiekol CHEMICAL CORPORATION
WASATCH DIVISION • BRIGHAM CITY, UTAH

Figure 3. Twelve Inch Bowl Vertical Slurry Friction Tester

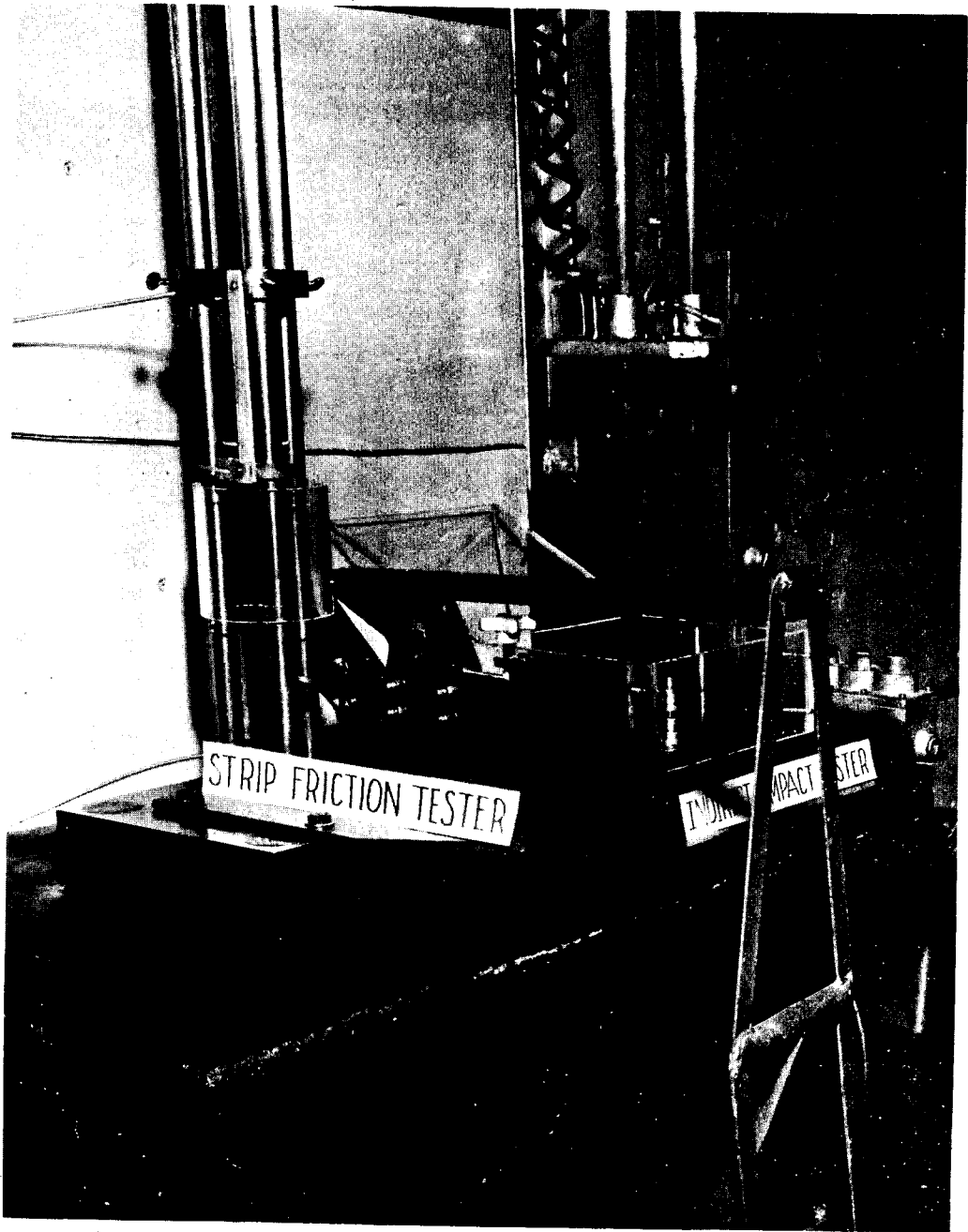


Thiekol CHEMICAL CORPORATION
WASATCH DIVISION • BRIGHAM CITY, UTAH

Figure 4. Twelve Inch Bowl Horizontal Slurry Friction Tester

STRIP FRICTION TEST SAMPLE & STRIPS

Thickol CHE Figure 5. Strips and Propellant Sample Used on Strip Friction Tester



Thiokol CHEMICAL CORPORATION
WASATCH DIVISION • BRIGHAM CITY, UTAH

Figure 6. Strip Friction Tester

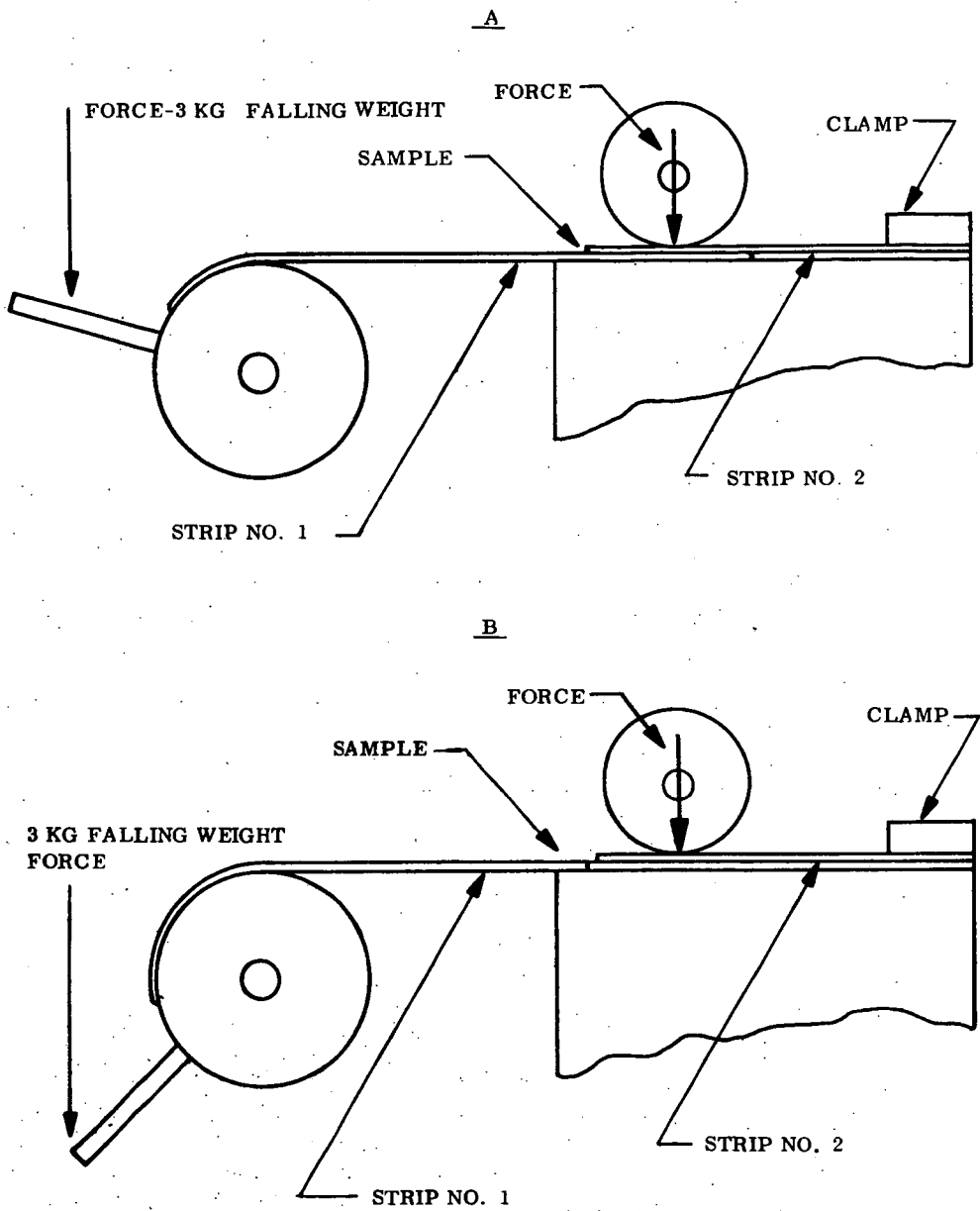


Figure 7. Detail of Strip Friction Tester

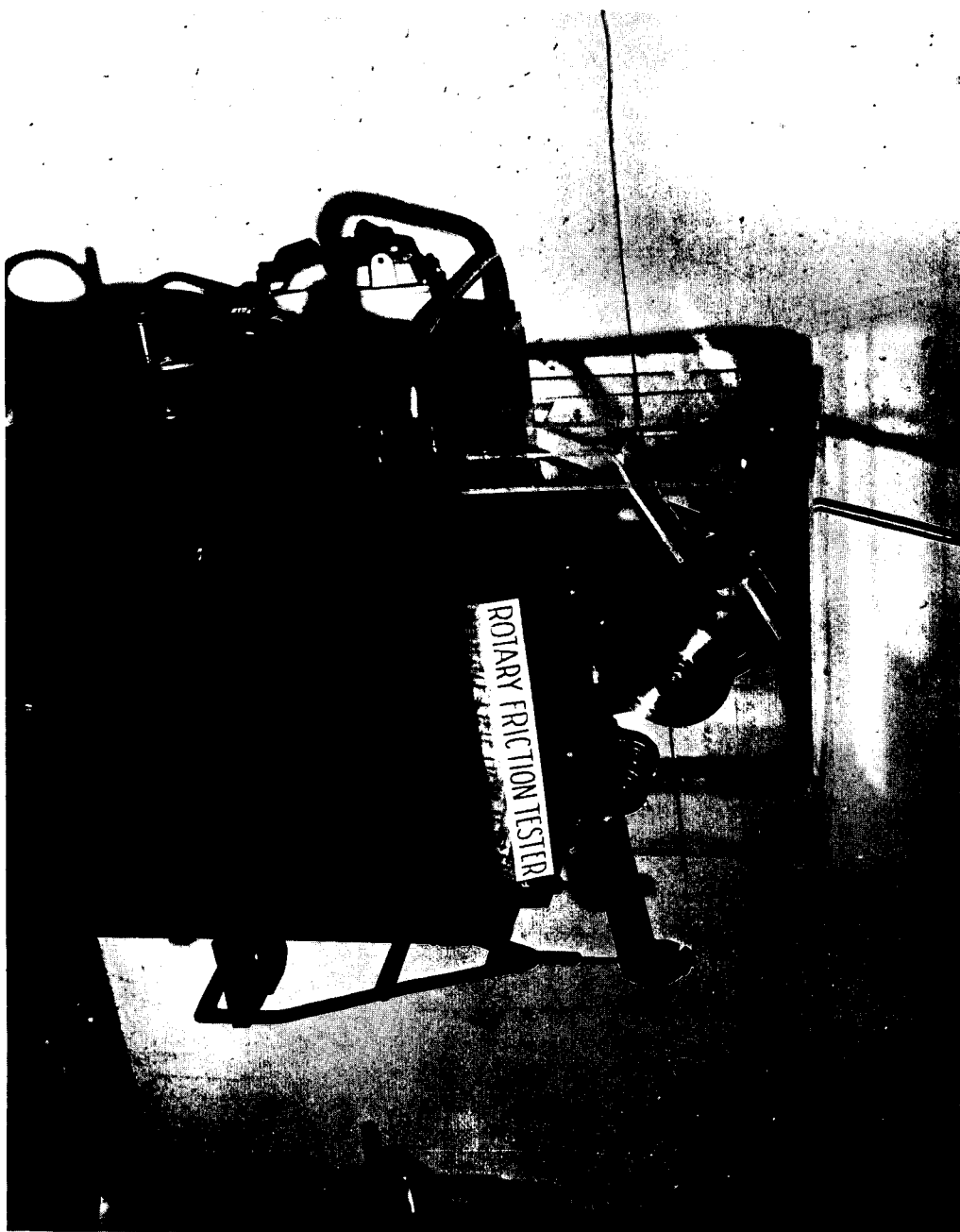
*Thiokol*CHEMICAL CORPORATION
WASATCH DIVISION • BRIGHAM CITY, UTAH

Figure 8. Rotary Friction Tester

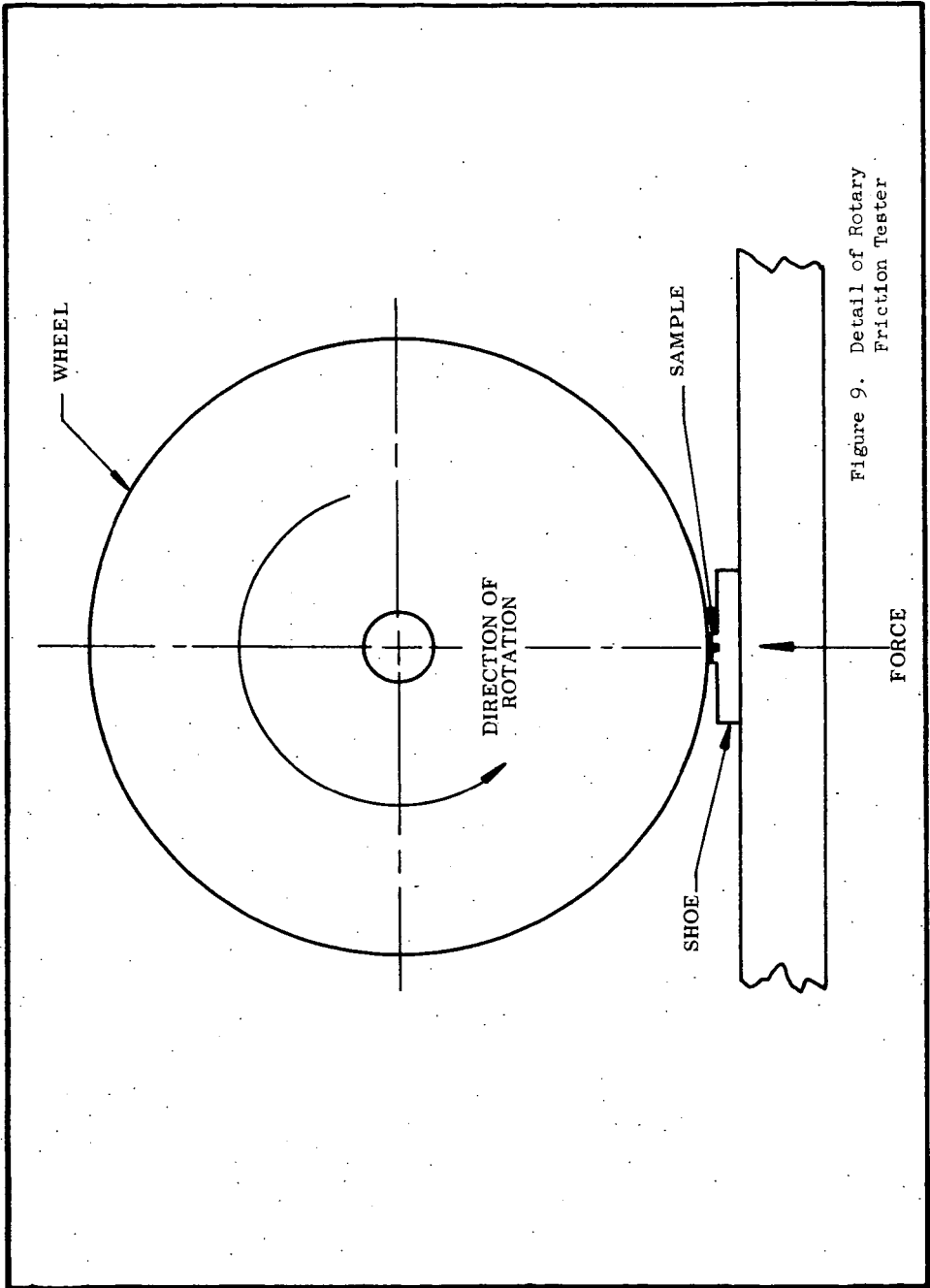
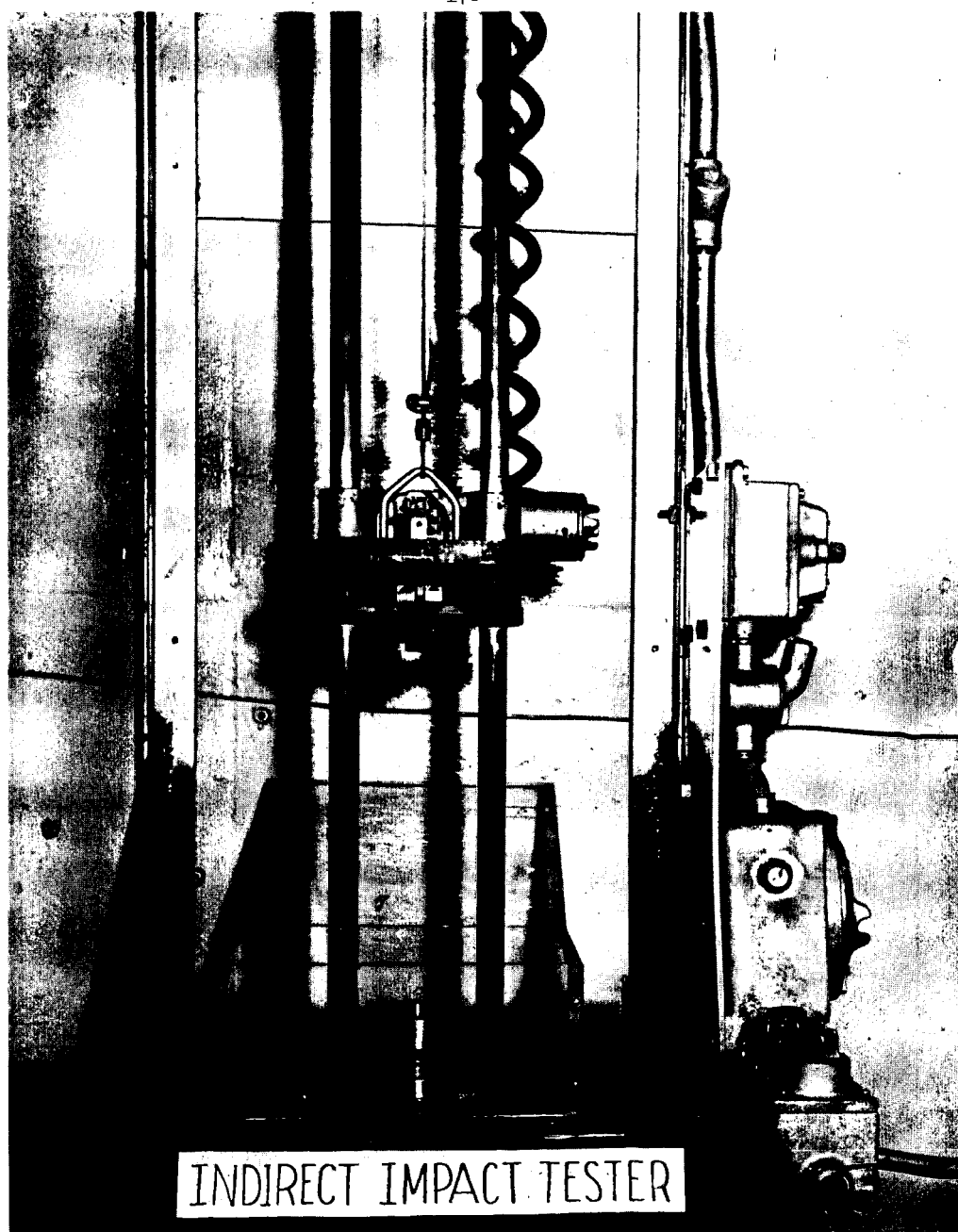


Figure 9. Detail of Rotary Friction Tester



Thickol WASATCH DIVISION • BRIGHAM CITY, UTAH

Figure 10. Indirect Impact Tester

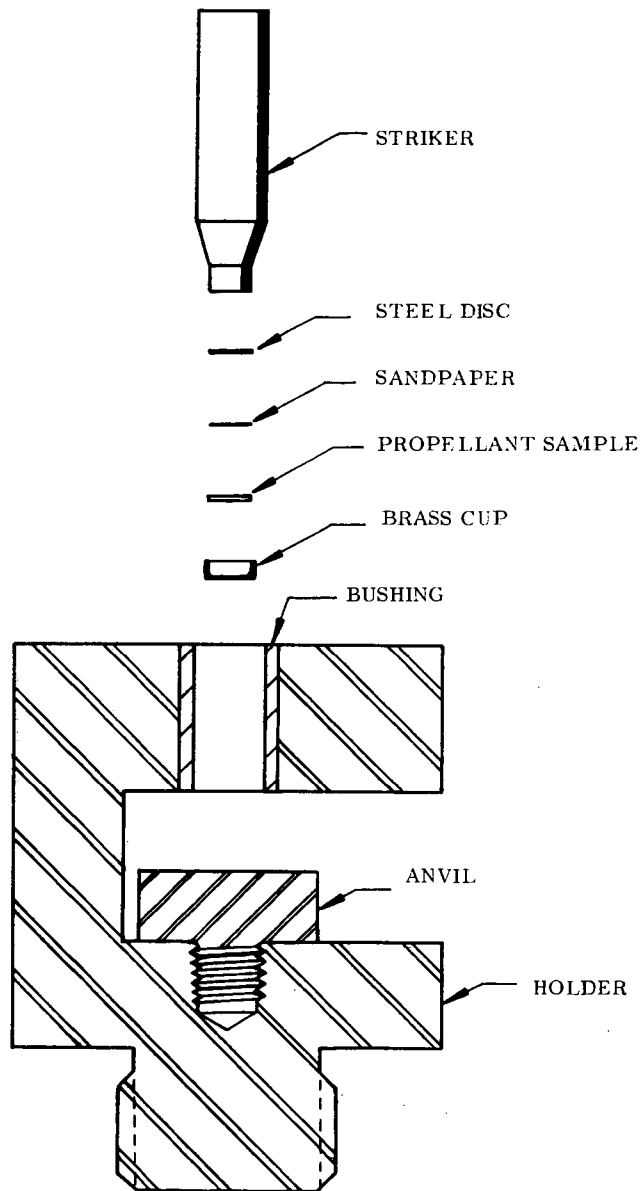


Figure 11

Details of Indirect Impact Tester & Assembly

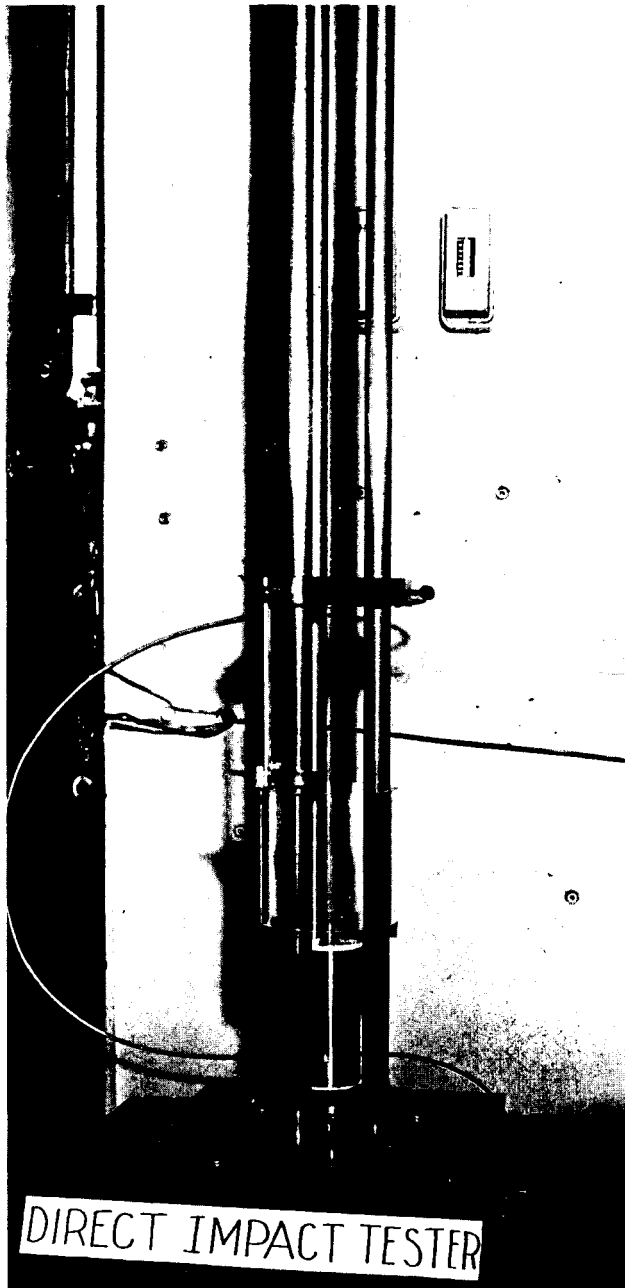


Figure 12. Direct Impact Tester

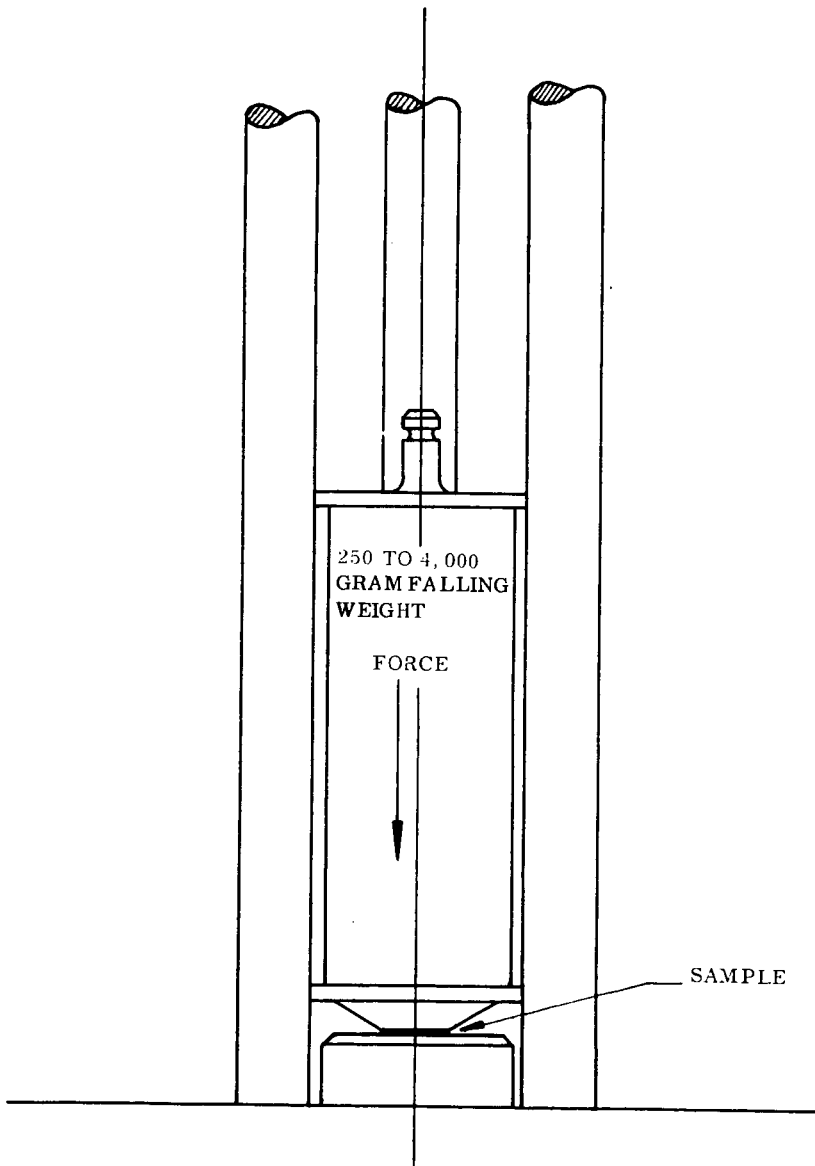
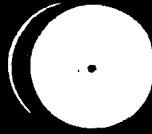


Figure 13

Striker Impacting on Sample
in Direct Impact Test

DIRECT IMPACT TEST SAMPLE & ANVIL



Thiokol

WILSON, J. C. & CO. INCORPORATED
WAXTON DIVISION • BRIDGE CITY, UTAH

Figure 14. Anvil and Propellant Sample Used on Indirect Impact Tester

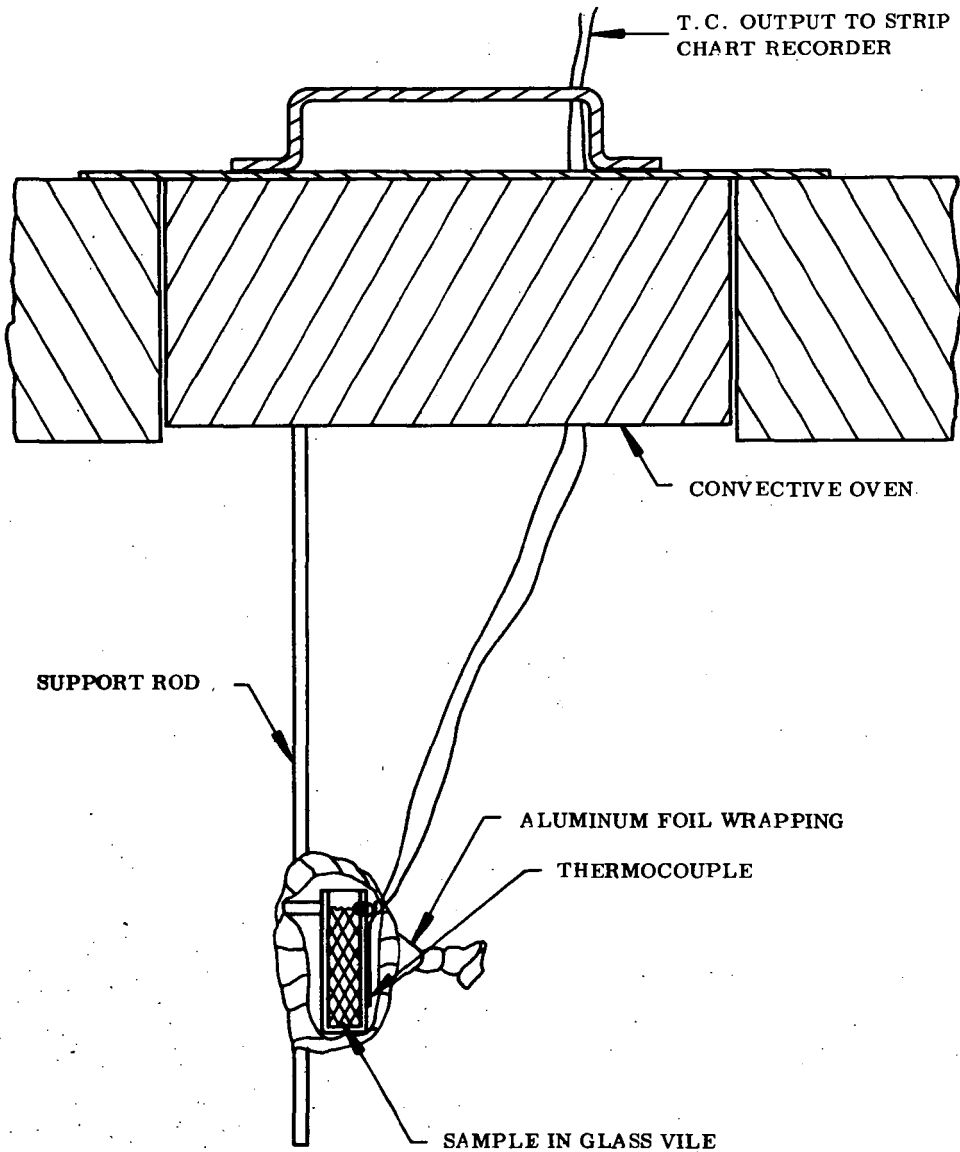
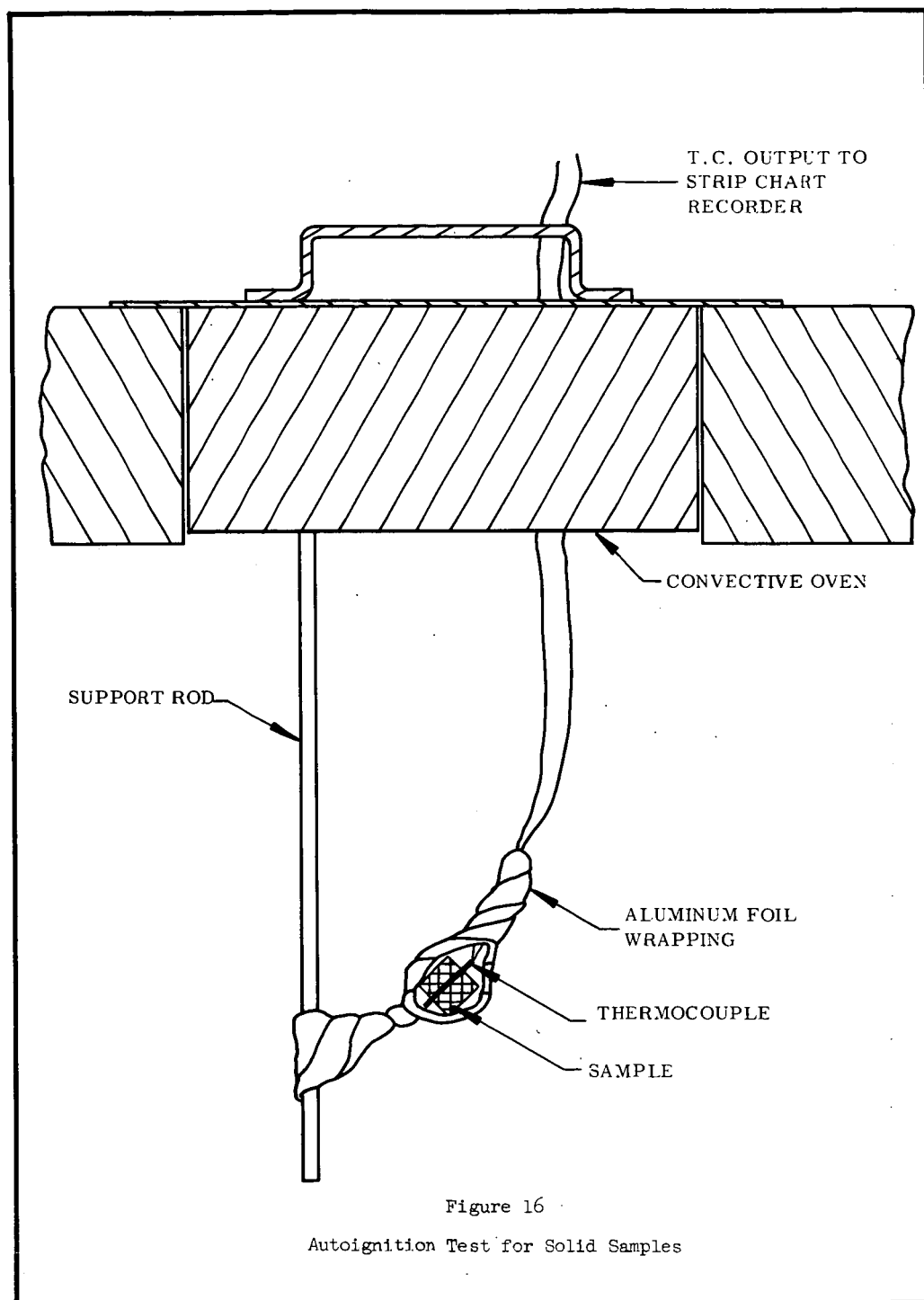
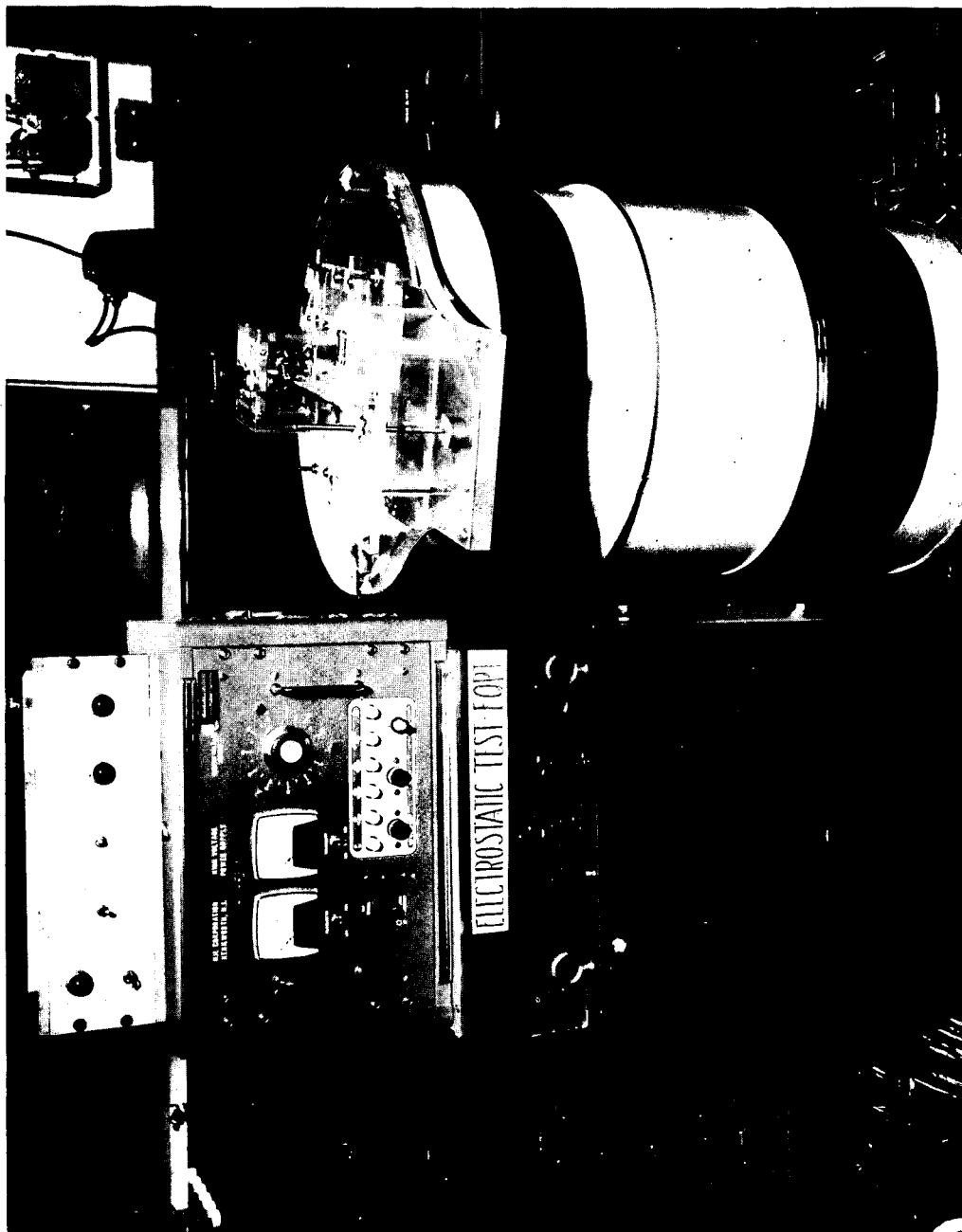


Figure 15

Autoignition Test for Liquid Samples





Thiokol

CHEMICAL CORPORATION
WATSON DIVISION • BELLGROVE, N.J. 07015

Figure 17. Electrostatic Test Equipment

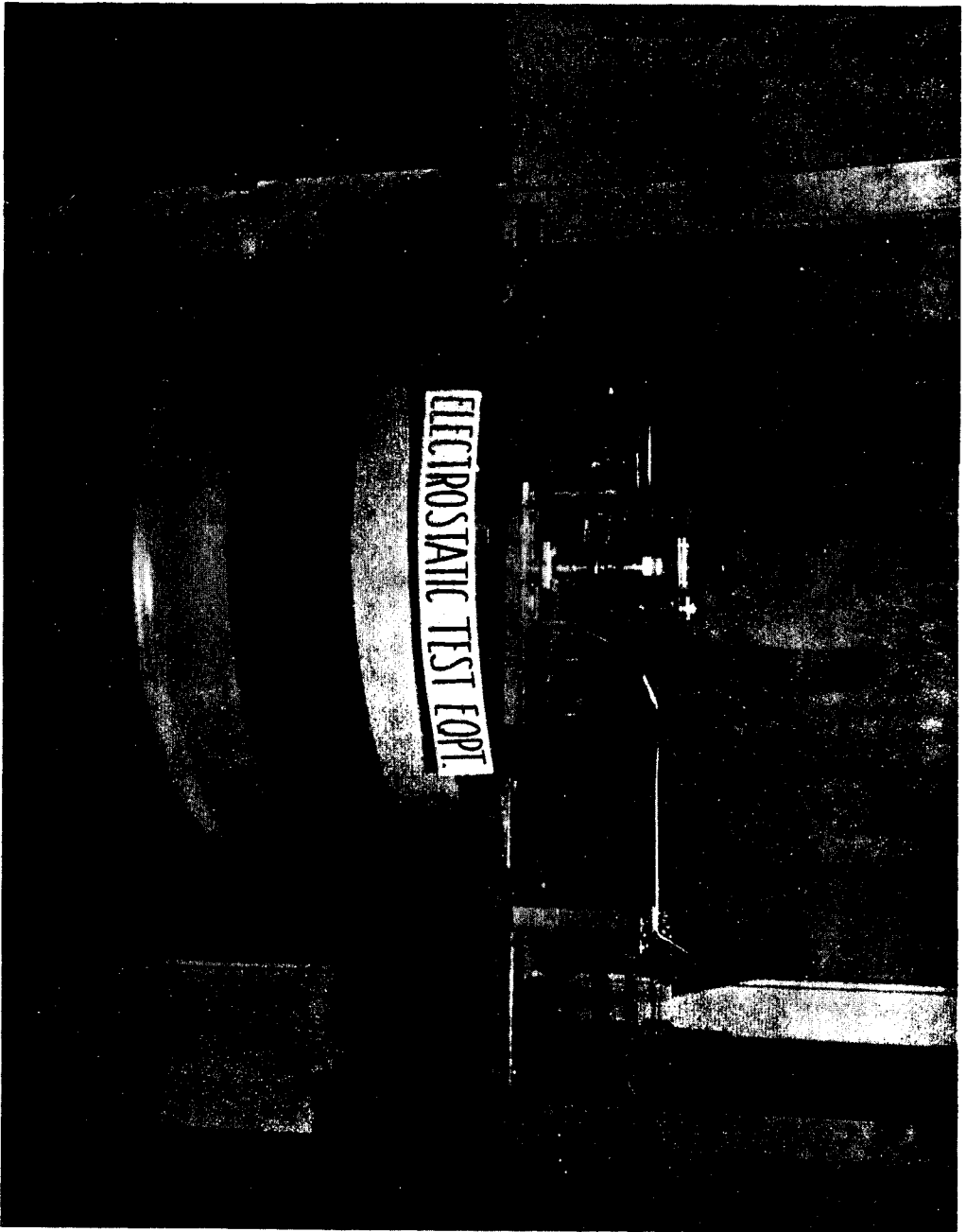


Figure 18. Sample Holder and Switch of Electrostatic Test Equipment

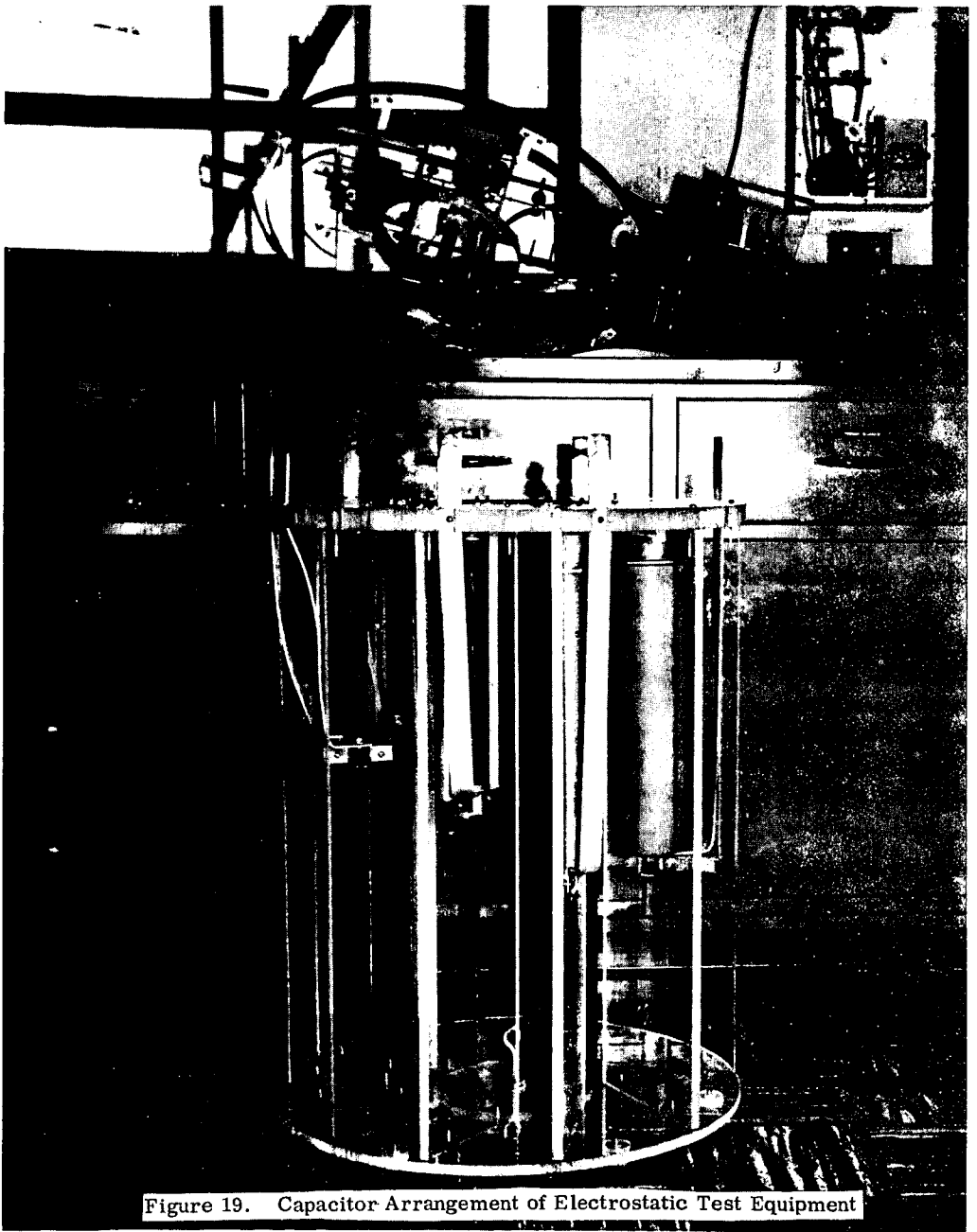


Figure 19. Capacitor Arrangement of Electrostatic Test Equipment

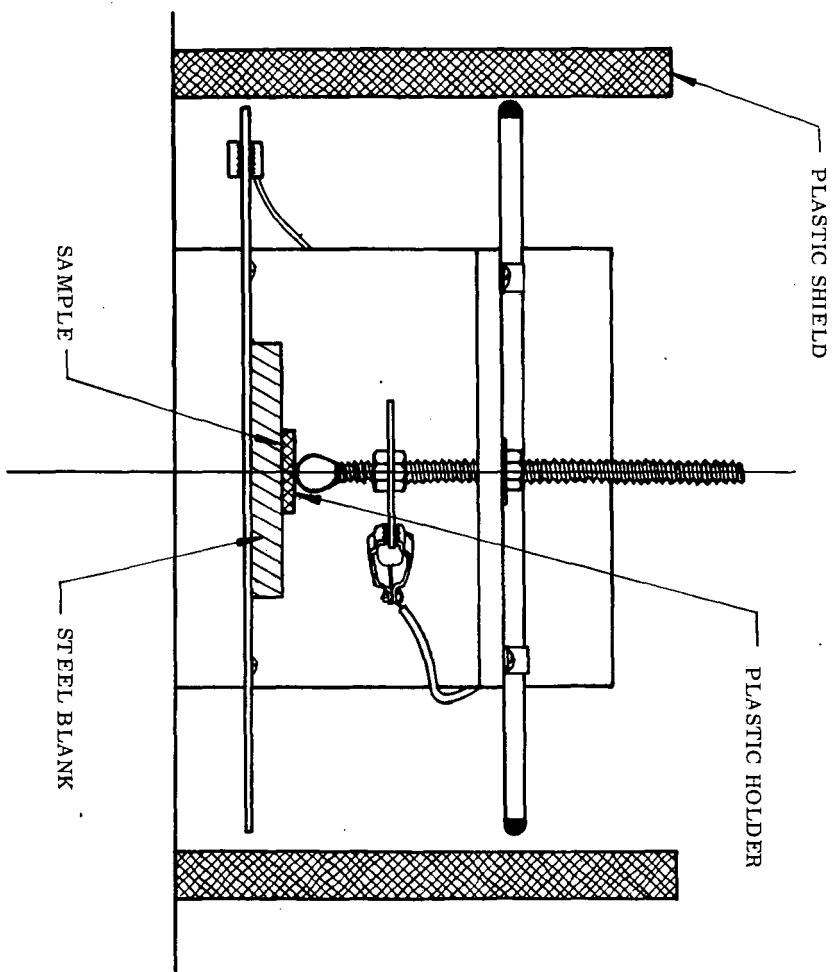


Figure 20. Electrostatic Test, Sample Test Fixture

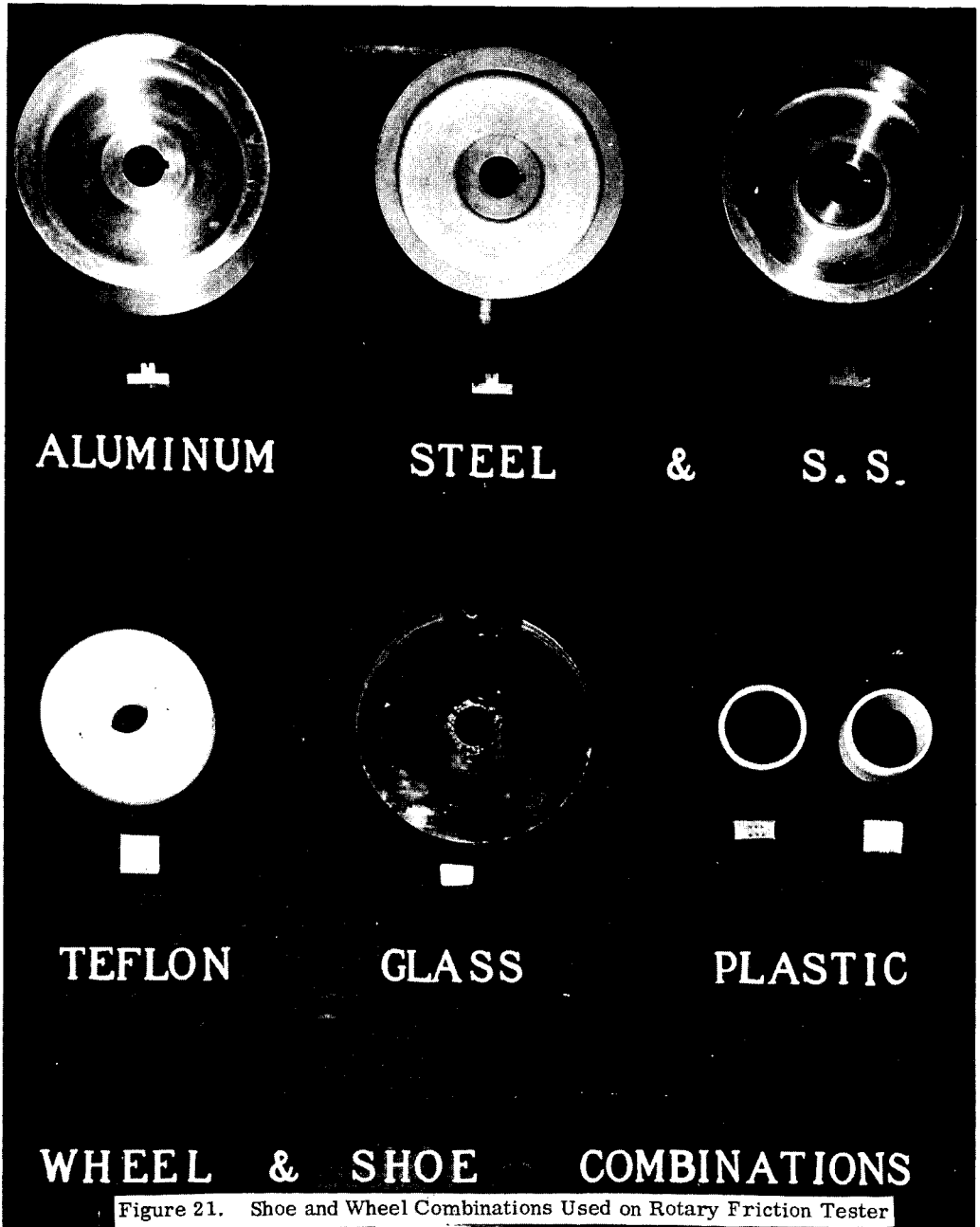


Figure 23. Typical Test Data Sheet

PROPELLANT SAFETY TESTS

| | | |
|--|--|---|
| | | PST NO. |
| DATE | WORK ORDER NO. | OPERATOR |
| THE FOLLOWING INDICATED SAFETY TESTS WERE CONDUCTED ON: | | |
| SAMPLE <div style="text-align: center;">001</div> | | EXPERIMENTAL <div style="text-align: center;">Y Propellant</div> |
| CONTAINING | | |
| IMPACT SENSITIVITY ESOP NO. 63-2 REV 3 | 50% EXPLOSION CORRECTED INCHES <div style="text-align: center;">12.8</div> | IMPACT INDEX <div style="text-align: center;">9.4</div> |

| DROP HEIGHT | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | RATIO | PERCENT |
|-------------|---|---|---|---|---|---|---|---|---|----|----|----|----|----|----|----|----|----|----|----|-------|---------|
| 16 | E | e | | e | e | | | e | | | E | e | | e | e | | e | | | | 10/10 | 100 |
| 14 | | E | | E | e | | | E | | N | | E | | E | e | | e | | | N | 8/10 | 80 |
| 12 | | | N | | E | | N | | N | n | | | N | | E | | E | | N | n | 3/10 | 30 |
| 10 | | | n | | | N | n | | n | n | | | n | | | N | | N | n | n | 0/10 | 0 |
| | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | |

COMMENTS



E = EXPLOSIONS
 e = ASSUMED EXPLOSIONS
 N = NO EXPLOSIONS
 n = ASSUMED NO EXPLOSIONS

DETONABILITY

 ESOP NO. 63-7 REV 1 THE SAMPLE ~~WAS~~ (DID NOT) DETONATE IN A 2 INCH CHARGE DIAMETER.

AUTOIGNITION

ESOP NO. 63-8 REV 1

| "F | 200 | 250 | 300 | 350 | 400 | 450 | 475 | 500 | 525 |
|-----|-----|-----|-----|------------------|-----|-----|-----|-----|-----|
| MIN | | | | No fire 24 hr | 64 | 25 | 18 | 15 | 6 |

NOTE: 1. A FIVE POUND DROP WEIGHT IS USED IN IMPACT TESTING.
 THE HMX STANDARD 50% EXPLOSION WAS 30 INCHES.

2. SENSITIVITY COMPARED WITH STANDARD PROPELLANT WITH AN ASSIGNED IMPACT INDEX OF 10. ANY SAMPLE WITH AN IMPACT INDEX LOWER THAN 5 IS IN THE CRITICAL RANGE AND EXTREME CARE SHOULD BE EXERCISED IN MANUFACTURE AND HANDLING.

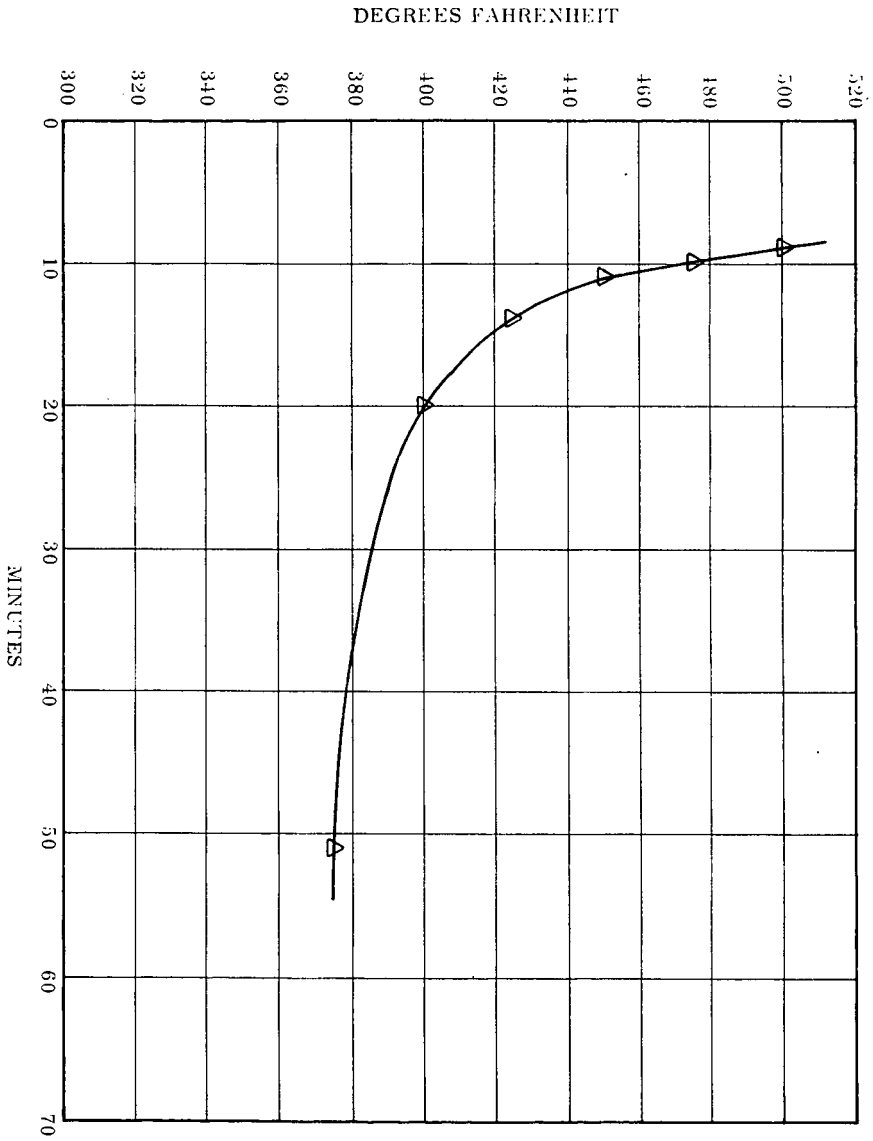


Figure 24. Avigation Test Curve

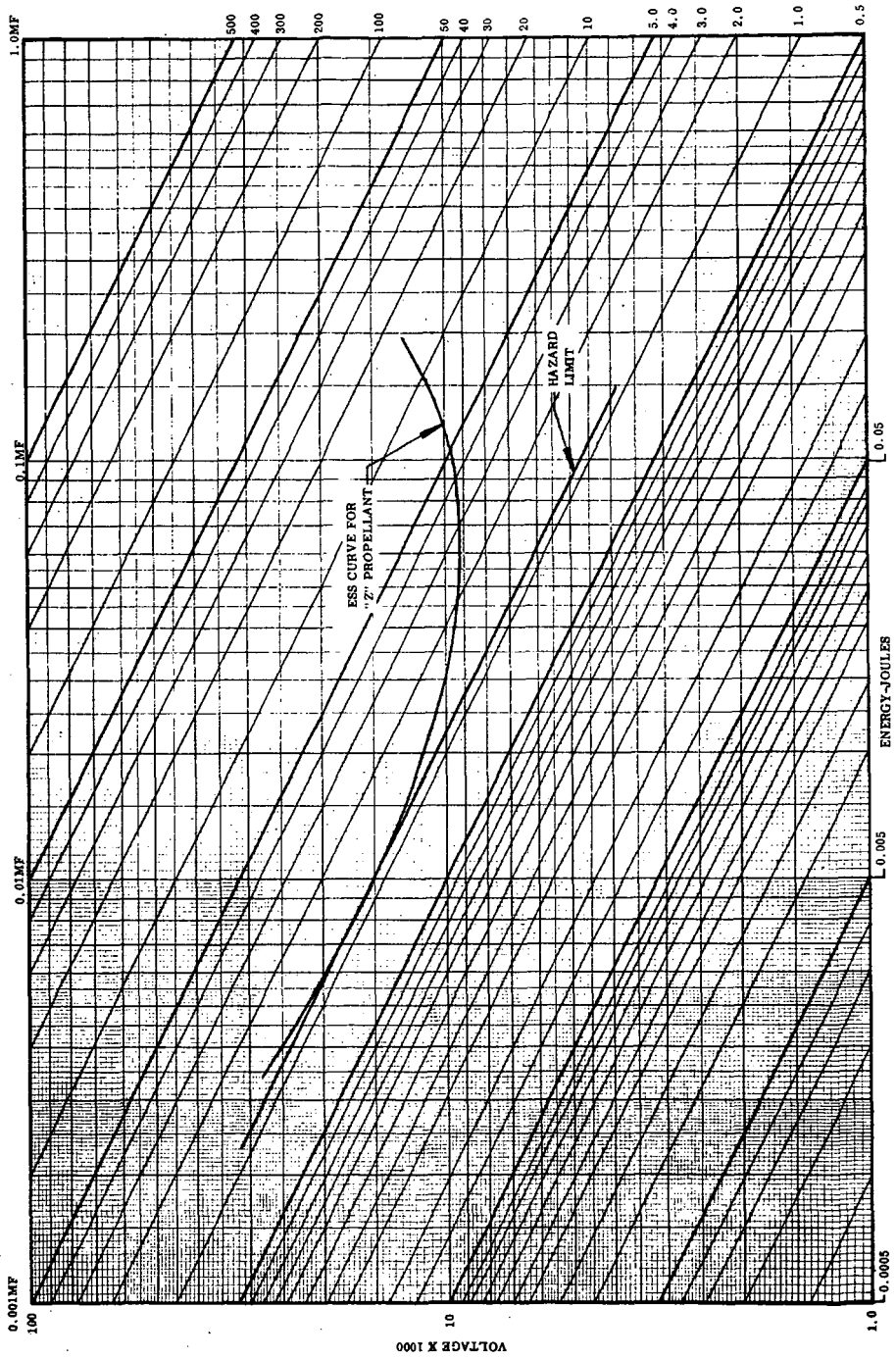


Figure 25. Chart of Voltages, Capacitance and Energy Levels

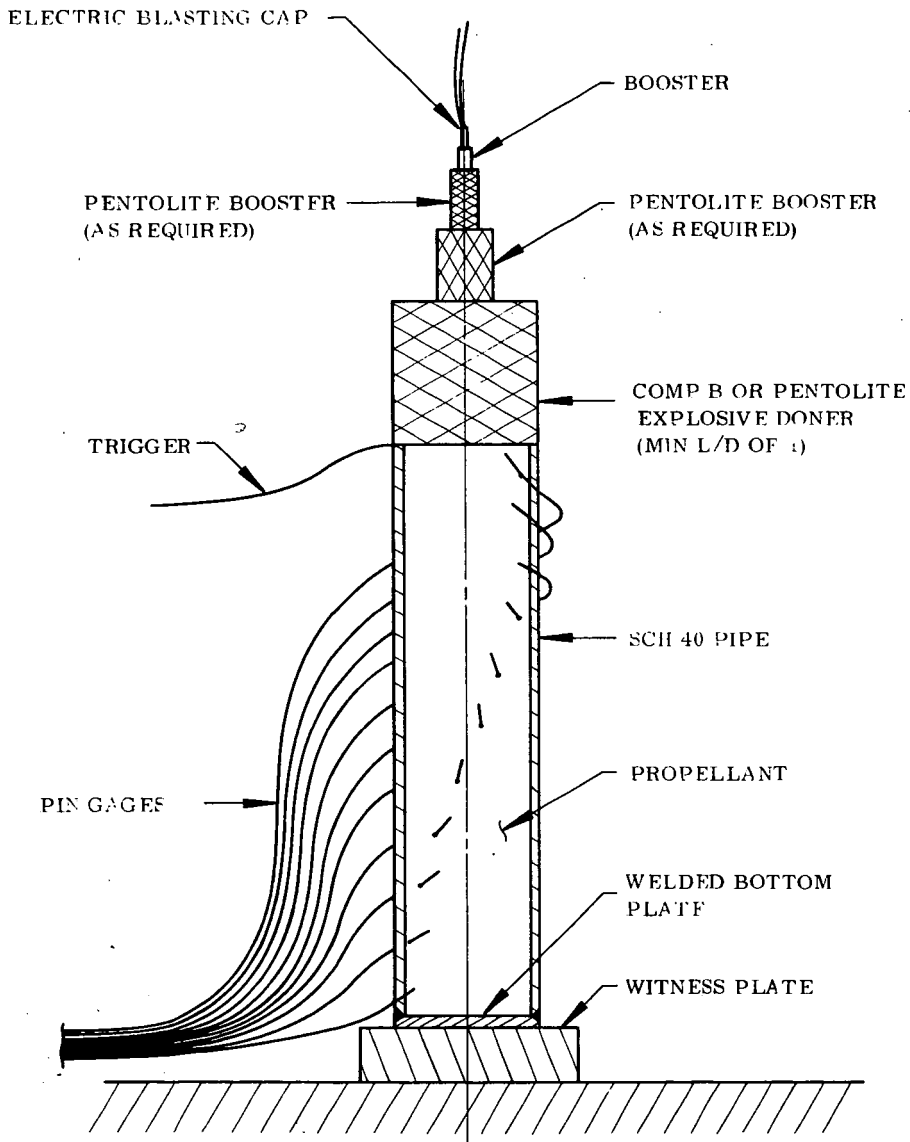


Figure 26. Standard Pipe Detonation Test Setup